phases, you do not have to control the temperature of the filter holder. Note that this differs from normal PM sampling procedures, which maintain the filter at a much lower temperature to capture a significant fraction of exhaust SVOC on the filter. In this method, SVOCs that pass through the filter will be collected on the downstream sorbent module. If you are collecting SVOCs in gas and particle phases, control your filter face temperature according to §1065.140(e)(4).

(3) Use good engineering judgment to design a cooling coil that will drop the sample temperature to approximately 5 °C. Note that downstream of the cooling coil, the sample will be a mixture of vapor phase hydrocarbons in CO_2, air, and a primarily aqueous liquid phase.

(4) Use a hydrophobic sorbent in a sealed sorbent module. Note that this sorbent module is intended to be the final stage for collecting the SVOC sample and should be sized accordingly. We recommend sizing the module to hold 40 g of XAD–2 along with PUF plugs at either end of the module, noting that you may vary the mass of XAD used for testing based on the anticipated SVOC emission rate.

(5) Include a condensate trap to separate the aqueous liquid phase from the gas stream. We recommend using a peristaltic pump to remove water from the condensate trap over the course of the test to prevent build-up of the condensate. Note that for some tests it may be appropriate to collect this water for analysis.

(d) *Sampler flow control.* For testing using the recommended filter and sorbent module sizes, we recommend targeting an average sample flow rate of 70 liters per minute to maximize SVOC collection. The sampler must be designed to maintain proportional sampling throughout the test. Verify proportional sampling after an emission test as described in §1065.545.

(e) *Water bath.* Design the sample system with a water bath in which the cooling coil, sorbent module, and condensate trap will be submerged. Use a heat exchanger or ice to maintain the bath temperature at (3 to 7) °C.

§1065.1107 Sample media and sample system preparation; sample system assembly.

This section describes the appropriate types of sample media and the cleaning procedure required to prepare the media and wetted sample surfaces for sampling.

(a) *Sample media.* The sampling system uses two types of sample media in series: The first to simultaneously capture the PM and associated particle phase SVOCs, and a second to capture SVOCs that remain in the gas phase, as follows:

(1) For capturing PM, we recommend using pure quartz filters with no binder. Select the filter diameter to minimize filter change intervals, accounting for the expected PM emission rate, sample flow rate, and number of repeat tests. Note that when repeating test cycles to increase sample mass, you may replace the filter without replacing the sorbent or otherwise disassembling the batch sampler. In those cases, include all filters in the extraction.

(2) For capturing gaseous SVOCs, utilize XAD–2 resin contained between two PUF plugs.

(b) *Sample media and sampler preparation.* Prepare pre-cleaned PM filters and pre-cleaned PUF plugs/XAD–2 as needed. Store sample media in containers protected from light and ambient air if you do not use them immediately after cleaning.

(1) Pre-clean the filters via Soxhlet extraction with methylene chloride for 24 hours and dry over dry nitrogen in a low-temperature vacuum oven.

(2) Pre-clean PUF and XAD–2 with a series of Soxhlet extractions: 8 hours with water, 22 hours with methanol, 22 hours with methylene chloride, and 22 hours with toluene, followed by drying with nitrogen.

(3) Clean sampler components, including the probe, filter holder, condenser, sorbent module, and condensate collection vessel by rinsing three times with methylene chloride and then three times with toluene. Prepare precleaned aluminum foil for capping the probe inlet of the sampler after the sampling system has been assembled.

(c) *Sorbent spiking.* Use good engineering judgment to verify the extent to which your extraction methods recover

SVOCs absorbed on the sample media. We recommend spiking the XAD–2 resin with a surrogate standard before testing with a carbon-13 or hydrogen-2 isotopically labeled standard for each of the class of analytes targeted for analysis. Perform this spiking as follows:

(1) Insert the lower PUF plug into the bottom of the sorbent module.

(2) Add half of one portion of XAD–2 resin to the module and spike the XAD–2 in the module with the standard.

(3) Wait 1 hour for the solvent from the standard(s) to evaporate, add the remaining 20 g of the XAD–2 resin to the module, and then insert a PUF plug in the top of the sorbent module.

(4) Cover the inlet and outlet of the sorbent module with pre-cleaned aluminum foil.

(d) *Sampling system assembly*. After preparing the sample media and the sampler, assemble the condensate trap, cooling coil, filter holder with filter, sample probe, and sorbent module, then lower the assembly into the reservoir. Cover the probe inlet with precleaned aluminum foil.

§ 1065.1109 Post-test sampler disassembly and sample extraction.

This section describes the process for disassembling and rinsing the sampling system and extracting and cleaning up the sample.

(a) *Sampling system disassembly*. Disassemble the sampling system in a clean environment as follows after the test:

(1) Remove the PM filter, PUF plugs, and all the XAD–2 from the sampling system and place them into a Soxhlet extraction thimble. Store them at or below 37 °C until analysis.

(2) Rinse sampling system wetted surfaces upstream of the condensate trap with acetone followed by toluene (or a comparable solvent system), ensuring that all the solvent remaining in liquid phase is collected (note that a fraction of the acetone and toluene will likely be lost to evaporation during mixing). Rinse with solvent volumes that are sufficient to cover all the surfaces exposed to the sample during testing. We recommend three fresh solvent rinses with acetone and two with toluene. We recommend rinse volumes of 60 ml per rinse for all sampling system components except the condenser coil, of which you should use 200 ml per rinse. Keep the acetone rinsate separate from the toluene rinsate to the extent practicable. Rinsate fractions should be stored separately in glass bottles that have been pre-rinsed with acetone, hexane, and toluene (or purchase pre-cleaned bottles).

(3) Use good engineering judgment to determine if you should analyze the aqueous condensate phase for SVOCs. If you determine that analysis is necessary, use toluene to perform a liquid-liquid extraction of the SVOCs from the collected aqueous condensate using a separatory funnel or an equivalent method. Add the toluene from this aqueous extraction to the toluene rinsate fraction described in paragraph (a)(2) of this section.

(4) Reduce rinsate solvent volumes as needed using a Kuderna-Danish concentrator or rotary evaporator and retain these rinse solvents for reuse during sample media extraction for the same test. Be careful to avoid loss of low molecular weight analytes when concentrating with rotary evaporation.

(b) *Sample extraction*. Extract the SVOCs from the sorbent using Soxhlet extraction as described in this paragraph (b). Two 16 hour extractions are necessary to accommodate the Soxhlet extractions of all SVOCs from a single sample. This reduces the possibility of losing low molecular weight SVOCs and promotes water removal. We recommend performing the first extraction with acetone/hexane and the second using toluene (or an equivalent solvent system). You may alternatively use an equivalent method such as an automated solvent extractor.

(1) We recommend equipping the Soxhlet extractor with a Dean-Stark trap to facilitate removal of residual water from the sampling system rinse. The Soxhlet apparatus must be large enough to allow extraction of the PUF, XAD–2, and filter in a single batch. Include in the extractor setup a glass thimble with a coarse or extra coarse sintered glass bottom. Pre-clean the extractor using proper glass-cleaning procedures. We recommend that the Soxhlet apparatus be cleaned with a (4